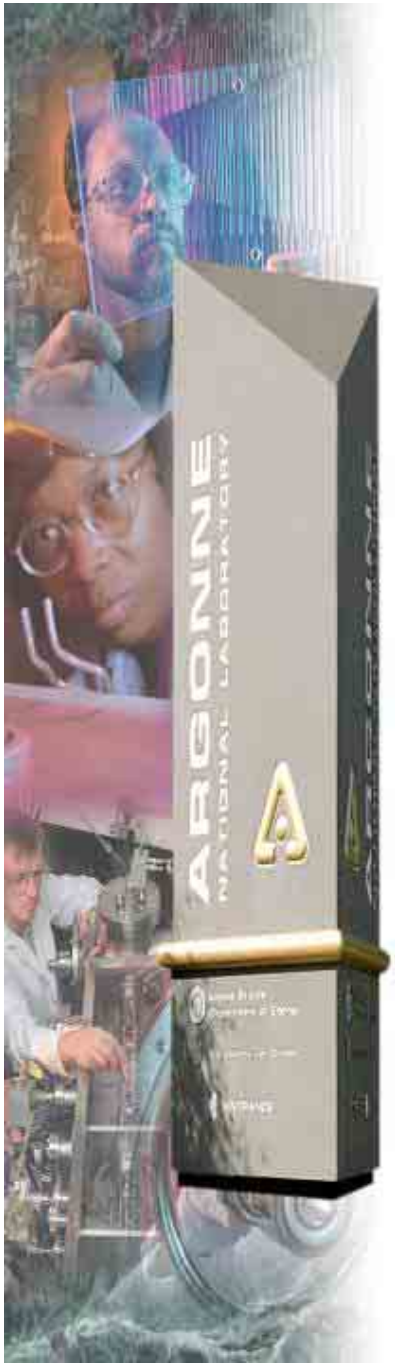


Development of Ultrananocrystalline Diamond (UNCD) Coatings

Presented by

Jeffrey Elam
Argonne National Laboratory

Materials, Sensors & Automation, and Glass Project Review
June 21-24, 2004
Arlington, Virginia

Office of Science
U.S. Department of Energy

A U.S. Department of Energy
Office of Science Laboratory
Operated by The University of Chicago

Project Summary

- **Goal:**
  - Use UNCD to achieve significant energy savings in IOF industries
    - *First application – SiC multipurpose mechanical pump seals*
- **Challenge:**
  - Develop technology to take UNCD from laboratory to market application
    - *Need to mass produce UNCD coated parts*
- **Benefits:**
  - Improved wear resistance and corrosion resistance of UNCD coated parts
  - 20% energy savings of 236 trillion Btu by 2020 in pump applications primarily due to reduced friction losses
- **FY05 Activities:**
  - Commission and optimize 11-inch IPLAS system (up from 6-inch system)
    - *Demonstrate UNCD coating of multiple seals simultaneously*
  - Demonstrate benefits of UNCD coatings on gas seals
  - Verify tribological benefits of UNCD coated seals
    - *Perform long-term pump tests*

Project Participants – Laboratory-led project

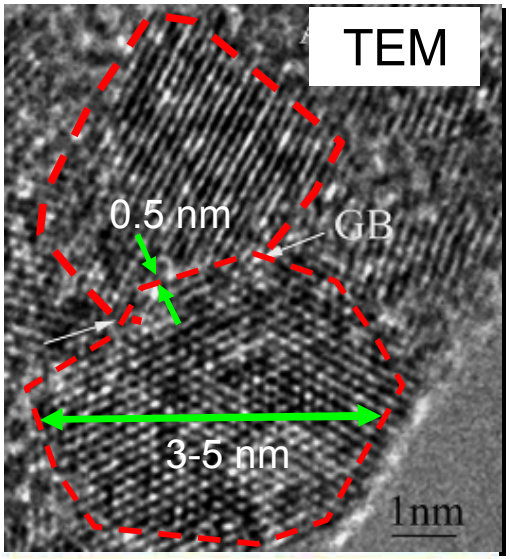
- **Argonne National Laboratory**
  - Energy Systems:
    - *Jeff Elam, John Hryn (Project POC), Joe Libera*
  - Energy Technology:
    - *Ali Erdemir, Andriy Kovalchenko*
  - Materials Science:
    - *Orlando Auciello, John Carlisle, Dieter Gruen, Mike Pellin, Alex Zinovev*

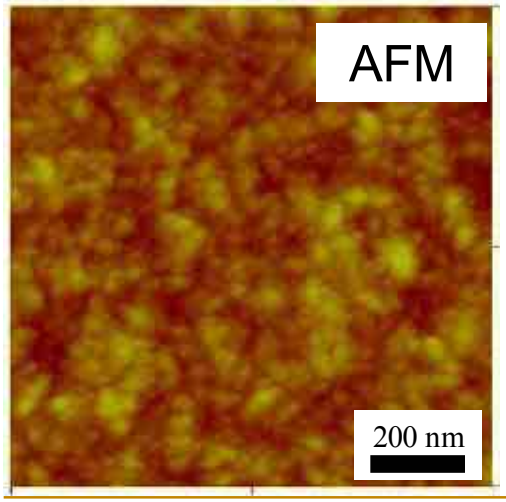
- **Industry Partners:**
  - Advanced Diamond Technologies, Inc. (ADT)
    - *Neil Kane (Industry POC)*
  - John Crane, Inc.
  - IPLAS Innovative Plasma Systems
  - Morgan Advanced Ceramics
  - Northwestern University
  - University of Illinois at Chicago

Barriers – Pathways – Critical Metrics

1) Uniform nucleation and growth of UNCD *1) smooth films* *2) good adhesion*	**Understand plasma deposition and surface seeding requirements, UNCD characterization and testing**	**Develop seeding protocol to produce uniform UNCD films (COMPLETED)**
2) Scale – up UNCD deposition process	**Commission 11-inch plasma system**	**Multiple seals coated with UNCD simultaneously**
3) Limited seal testing facilities	**Use industrial facilities and expand lab capabilities**	**Perform successful pilot test (data indicate energy savings)**
4) Commercialization of UNCD technology	**Launch company (ADT)**	**Sign toll-processing agreement**

Ultrananocrystalline Diamond (UNCD) Properties





- Hardness - 97 GPa
- Elastic Modulus - 970 GPa
- Fracture Strength - 5 GPa

- Grain Size - 2 - 5 nm
- RMS Roughness - 0.5 - 1 μ-inch
- Friction Coefficient - 0.03

UNCD for Multipurpose Mechanical Pump Seals

UNCD Coating

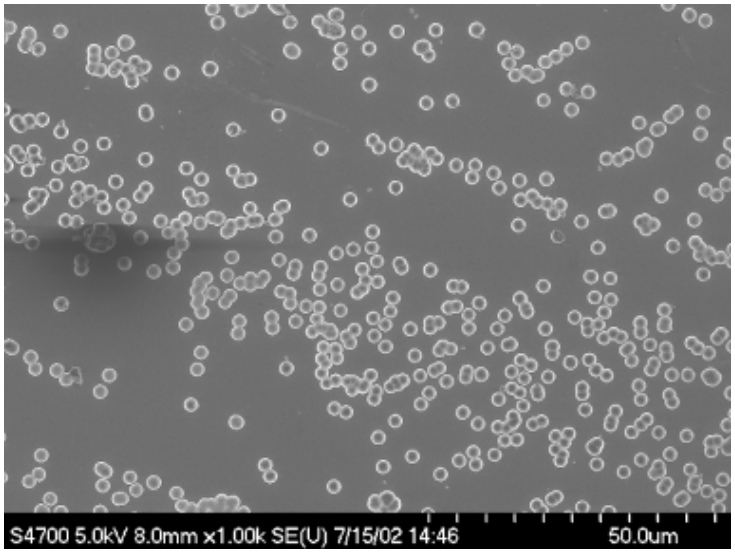
- **UNCD to reduce friction and eliminate wear**

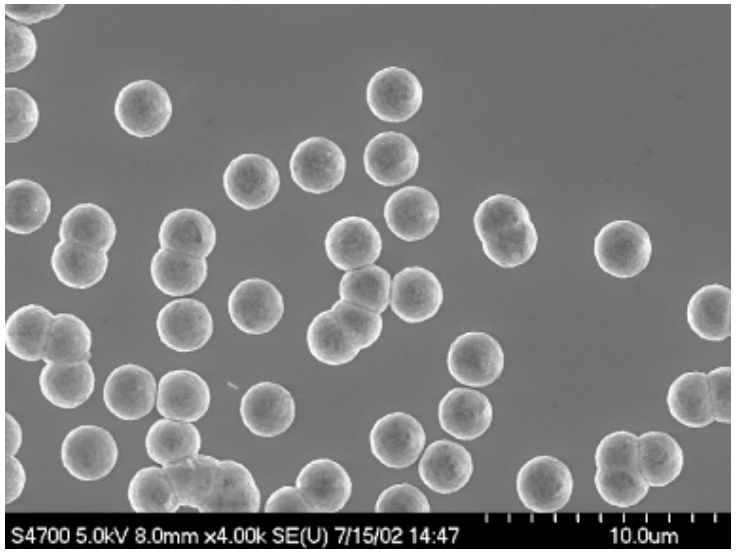
Technical Barrier #1: Uniform nucleation and growth of UNCD

- **Seeding to achieve smooth UNCD films**
- **Seeding to achieve excellent adhesion of UNCD to SiC Seal**

UNCD – Example of Poor Seeding

Sample: Unseeded CVD SiC

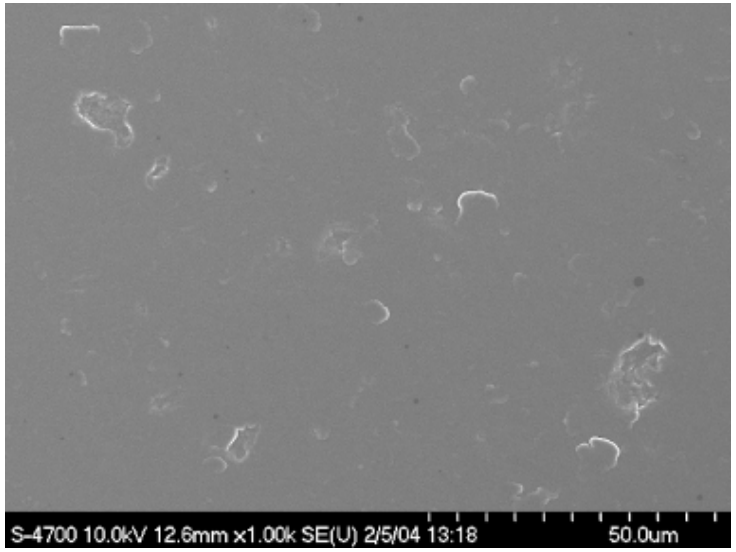


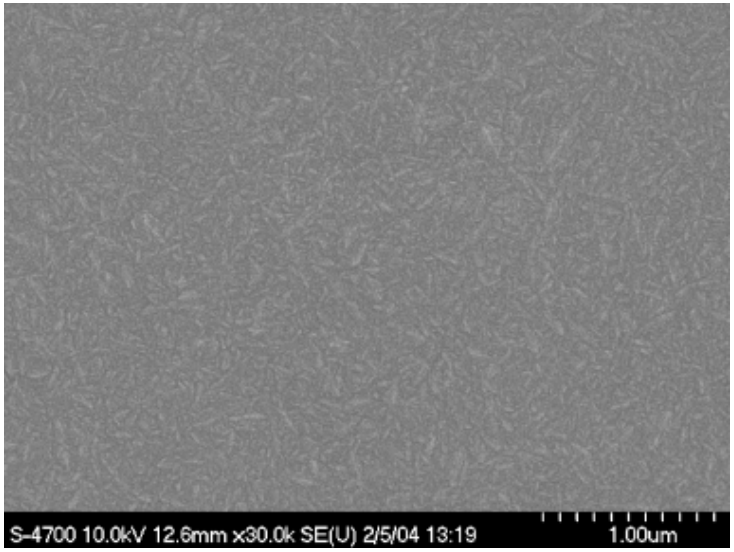


- Low nucleation density – discontinuous coating

UNCD – Example of Excellent Seeding

Sample: Mechanically Seeded α-SiC

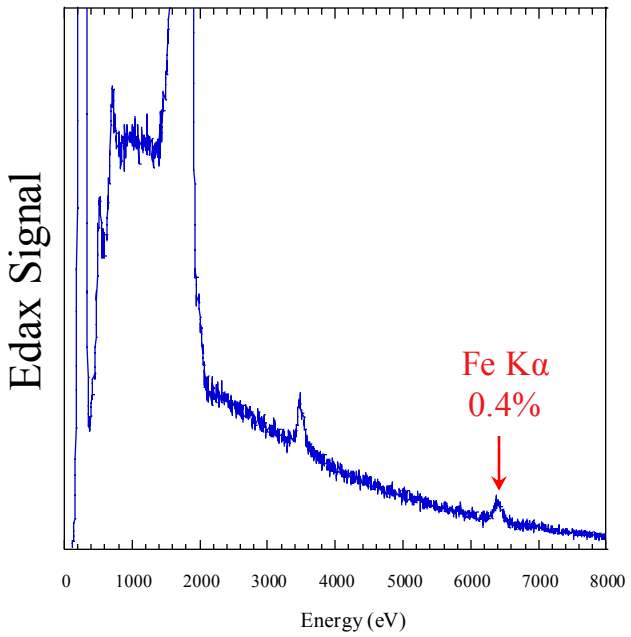




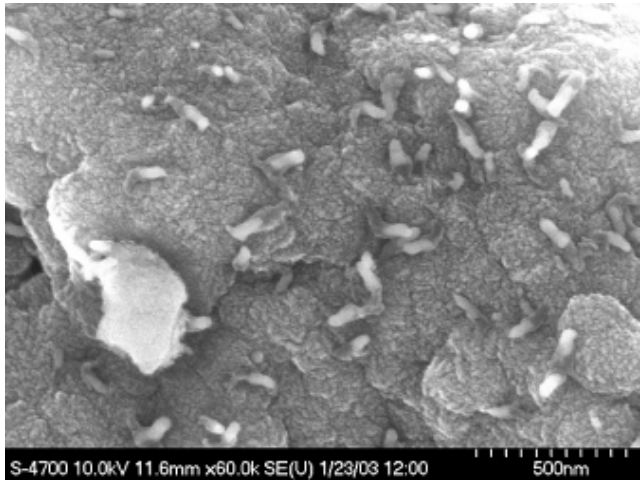
- High nucleation density - dense, continuous, smooth film

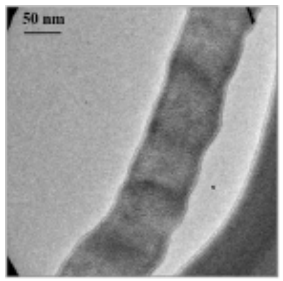
Simultaneous UNCD and Carbon Nanofiber (CNF) Growth

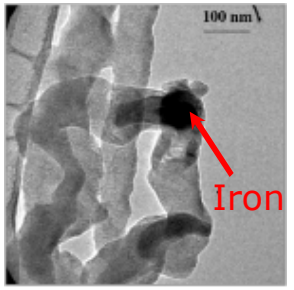
EDAX Elemental Analysis



SEM and TEM Following UNCD Treatment



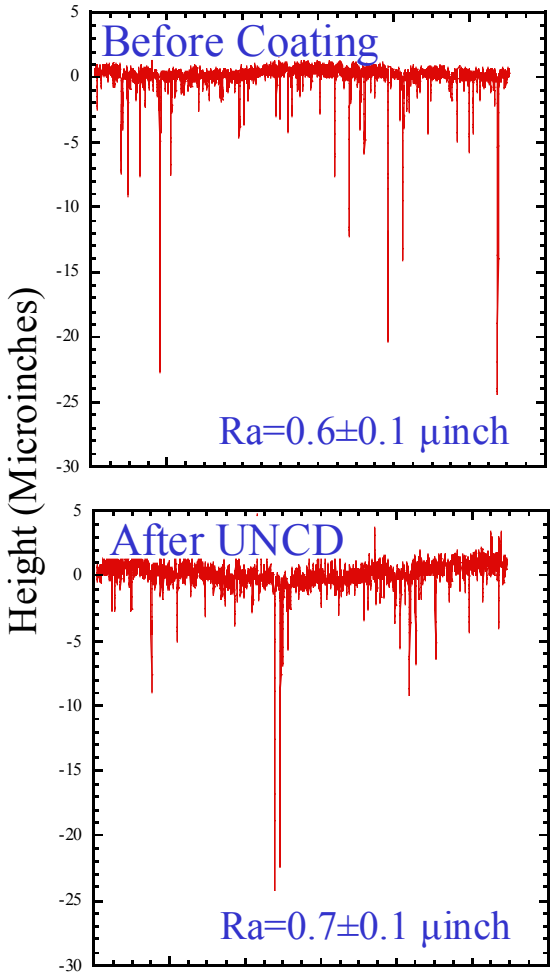




SEM, TEM shows CNF with iron particle catalyst

- Trace Fe Contamination Catalyzes CNF Growth
- Developed Screening Process for Iron

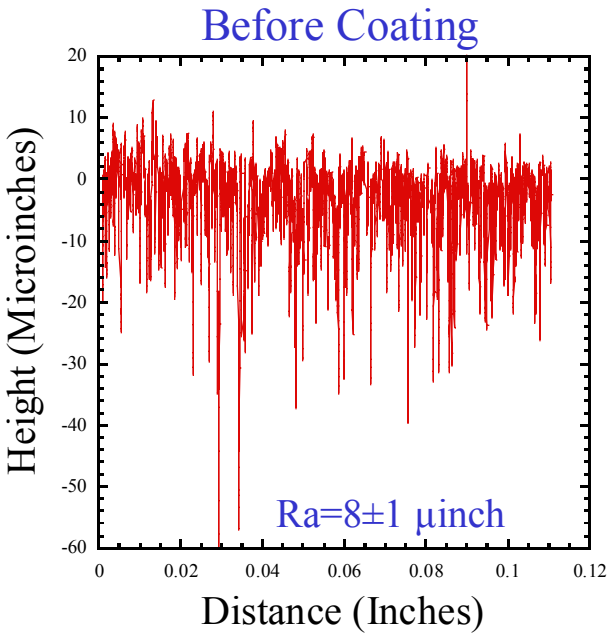
UNCD Coating of Smooth 2” OD α-SiC Seals



Pioneering Science and Technology

- UNCD is smooth, no roughness change from UNCD coating

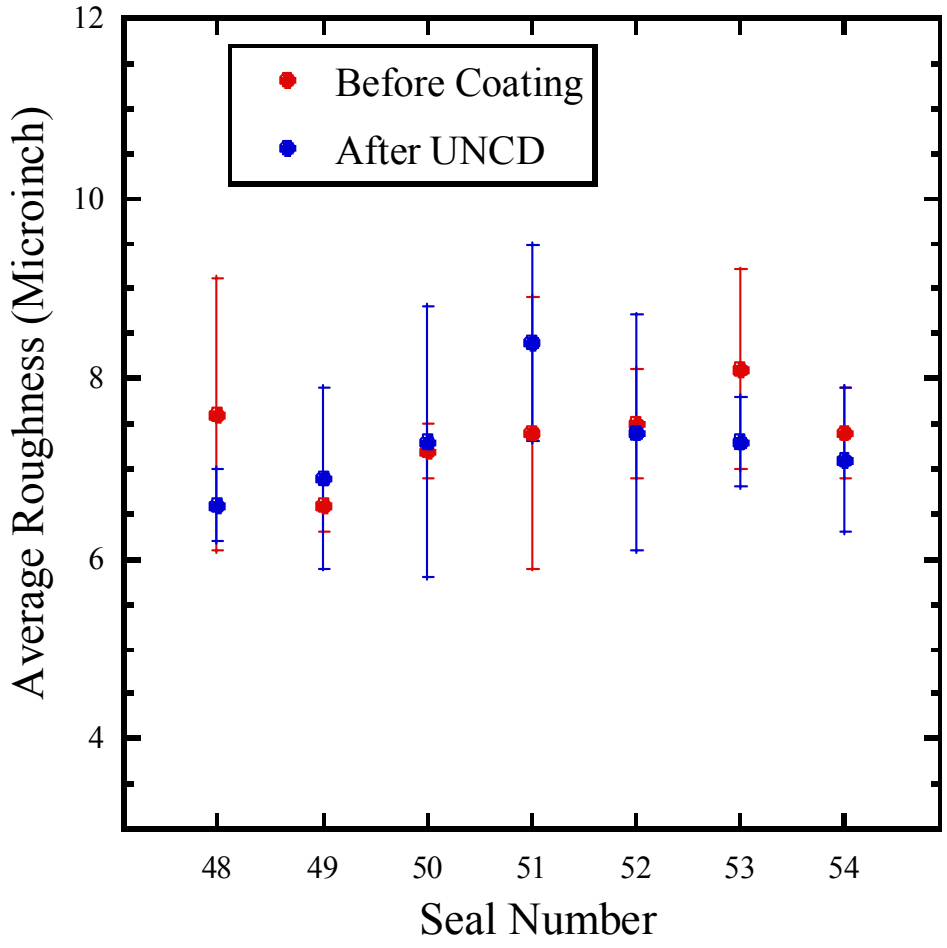
UNCD Coating of Rough 1” OD α-SiC Seals



Pioneering Science and Technology

- Rough initial surface for 1” seals

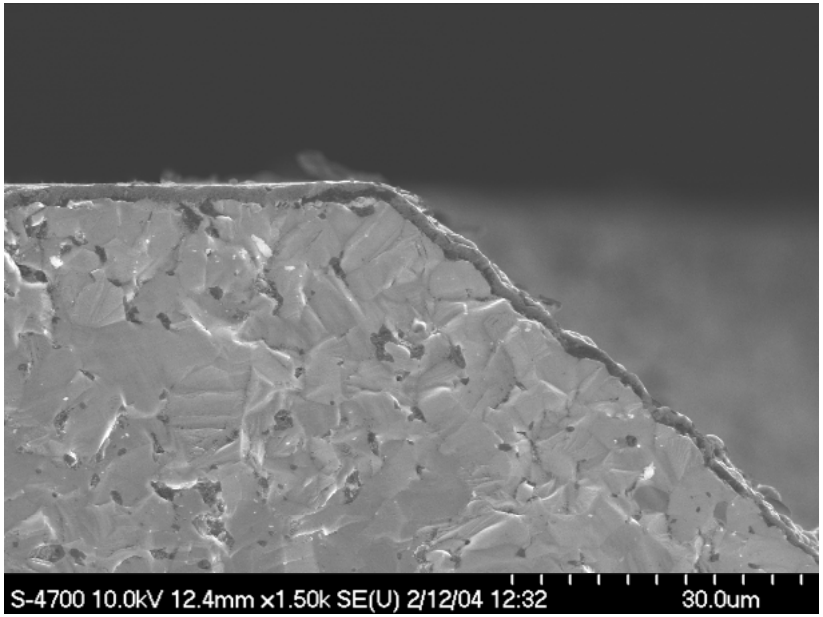
Roughness Measurements of Rough 1” OD Seals Following UNCD Coating



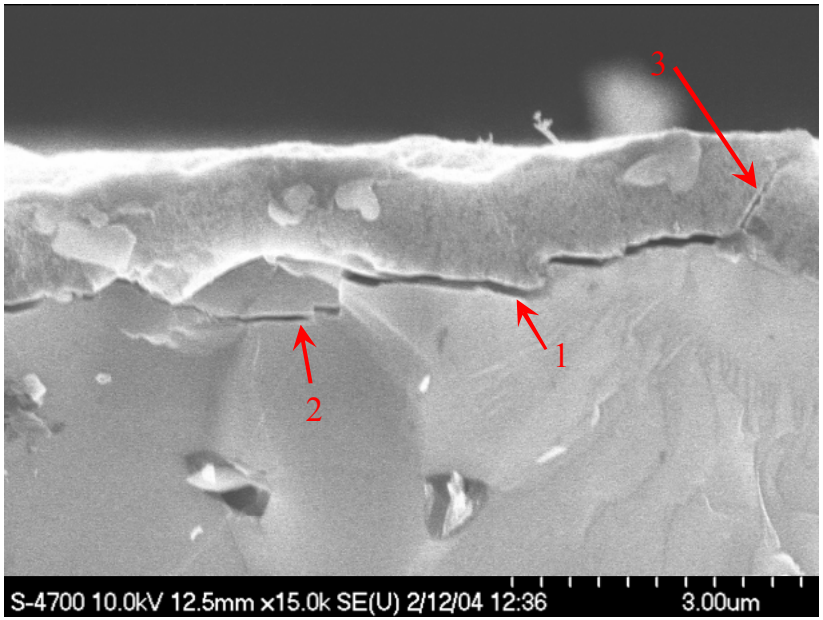
Pioneering Science and Technology

- No roughness change from UNCD Coatings

Adhesion Measurements Using Fracture Analysis



Conformal UNCD coating over
Seal face and beveled edge

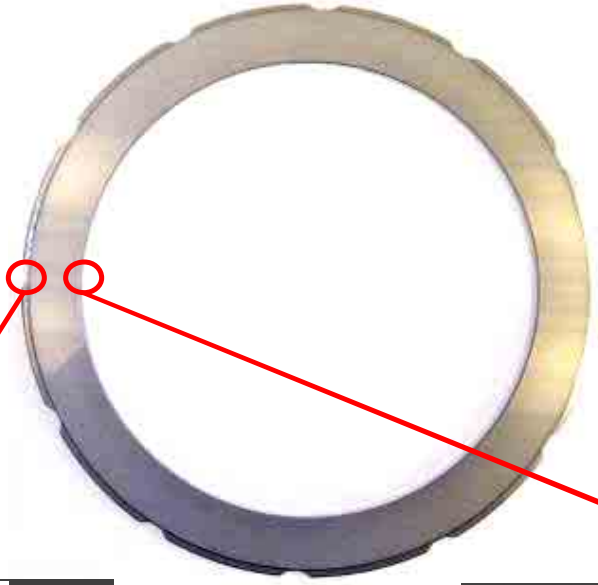


Fracture of UNCD-SiC interface observed after diamond saw cutting (1). However, fracture also observed along SiC grains (2) and UNCD film (3)

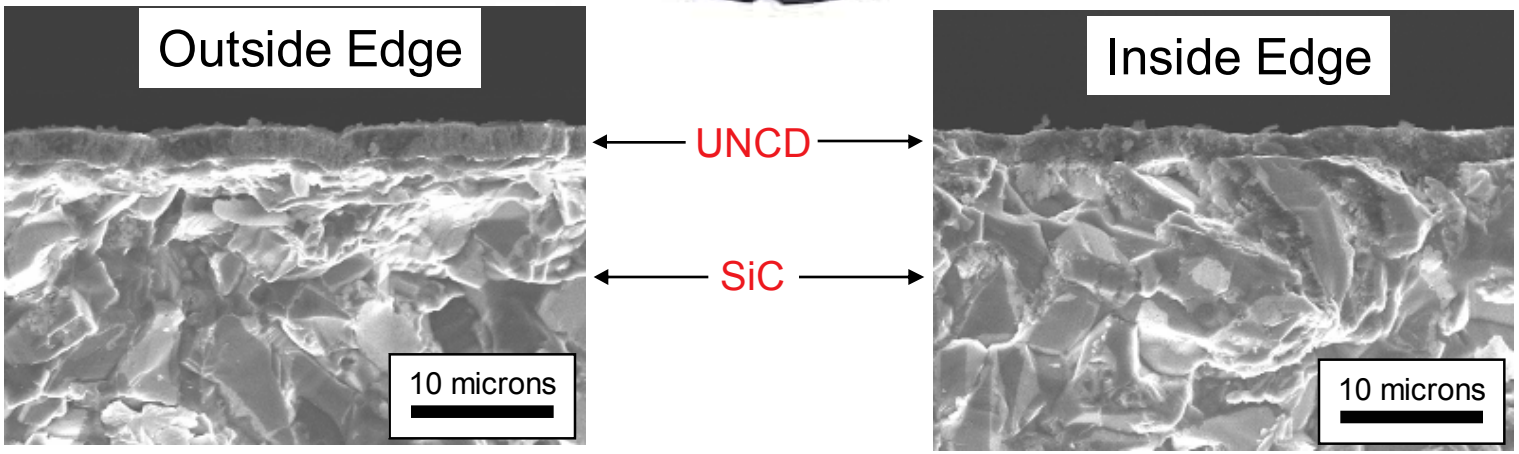
- Strong adhesion of UNCD Coating to SiC Surface

UNCD Coating of 5” Gas Seals

- Improved coating technology required for 5” gas seals
- Coating must preserve precisely engineered taper



- Segment of 5” seal was coated in existing small-area plasma system



- UNCD Maintains Critical Tolerance Across Face of 5” Gas Seal

Technical Barrier #2: Scale – up UNCD deposition process

IPLAS 11" Microwave Plasma CVD System

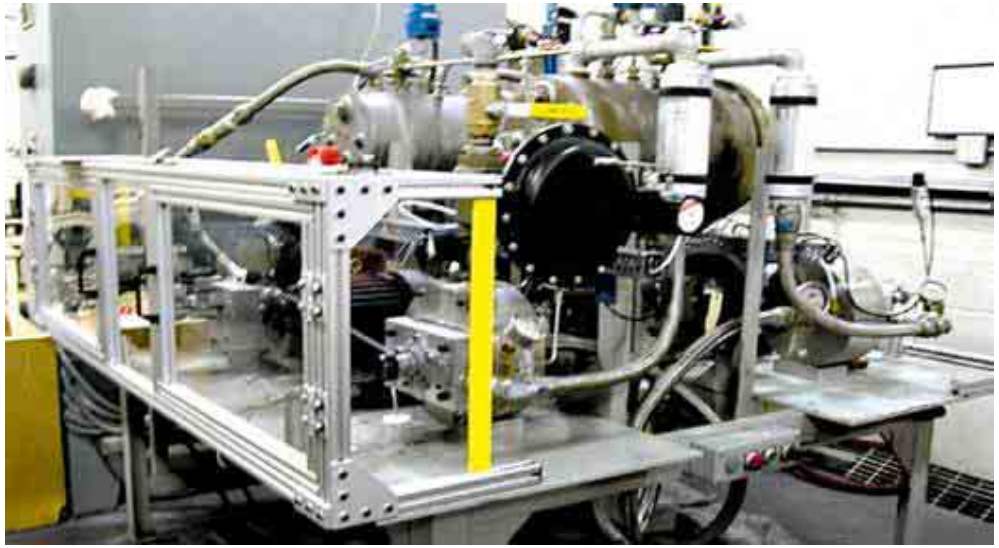
- First unit of its kind in the world
- Will enable batch-coating of multiple 2" seals
- Will enable coating of intact 5" seals

- Work is underway to install and commission this system

Technical Barrier #3: Limited seal testing facilities

- **Building Test Pump Loop at Argonne for Measuring Seal Friction and Wear**
- **Installed Surface Profiler at Argonne**
- **Friction and Wear Analysis at John Crane Testing Facilities**

Hot Water Test of UNCD-Coated Seals at John Crane

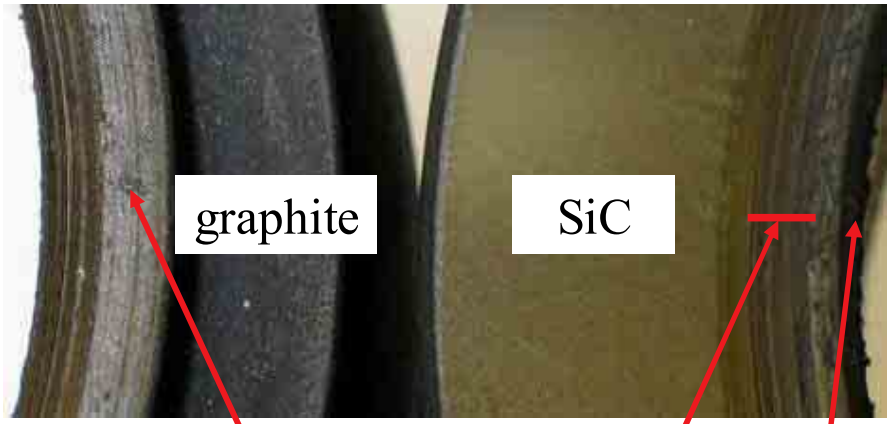


- 2” OD Seal
- 100 Hours

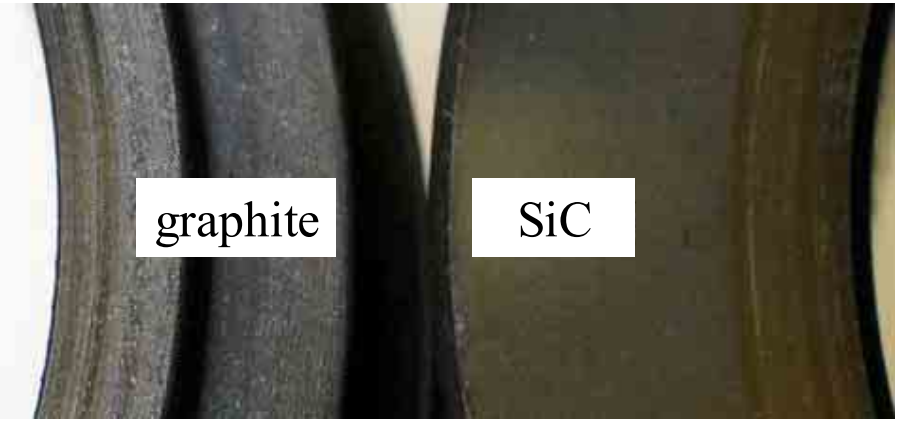
- Very aggressive test of materials properties
- Mimics harsh conditions in chemical process pump
- 100 hour test simulates 2 years of extreme use

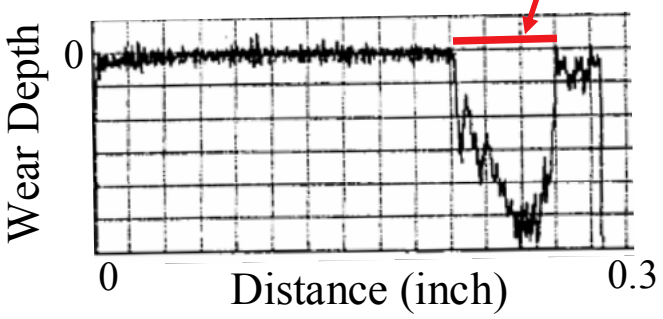
Hot Water Test of UNCD-Coated Seals

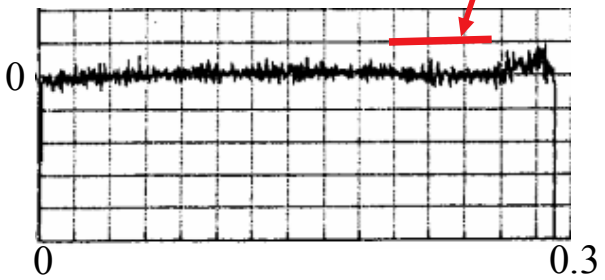
Uncoated



UNCD-Coated



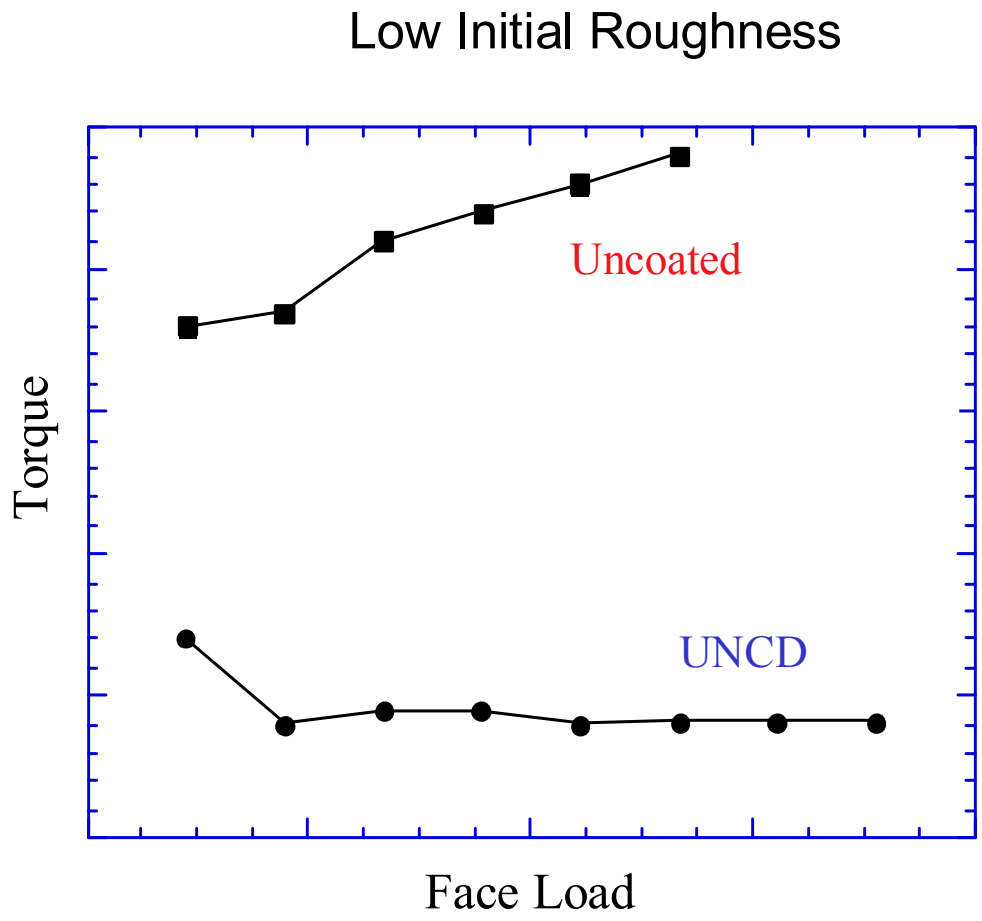




Pioneering Science and Technology

- Failure of uncoated seal, no detectable wear for UNCD-coated seal

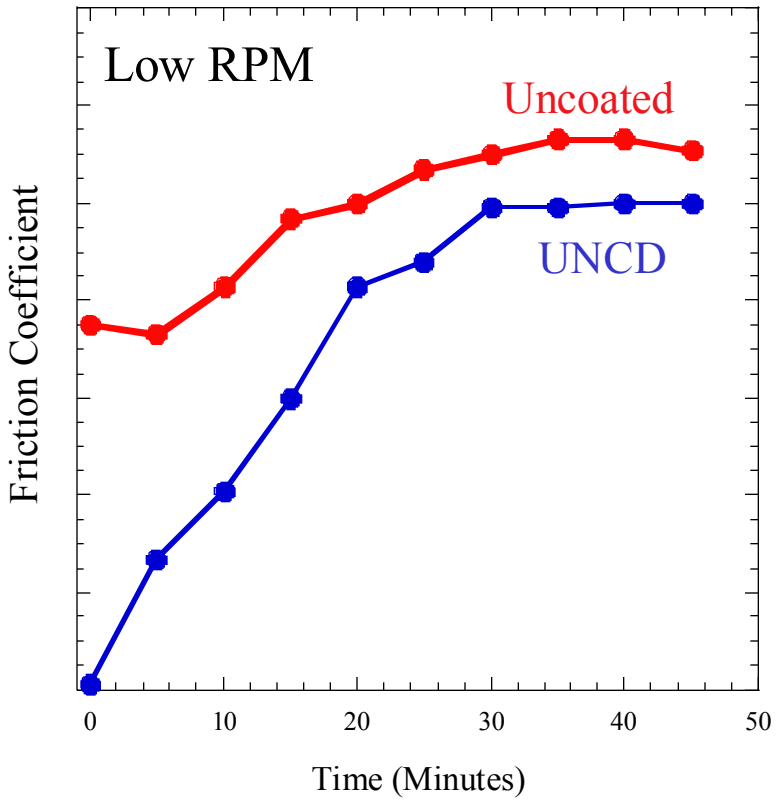
Friction Measurements of UNCD Coated 2” SiC Seal



- Significantly Reduced Friction for Smooth Seal Surface

Friction Measurements of UNCD Coated 1” SiC Seal

High Initial Roughness



- Marginally Reduced Friction for Rough Seal Surface

Pioneering Science and Technology

➡ Current Study: Effect of Roughness on UNCD Friction

Technical Barrier #4: Commercialization of UNCD technology

- **Advanced Diamond Technologies**
  - Argonne-initiated start-up company
    - *Exclusive license in UNCD application*
  - Company officially launched in 2003
  - Business plan established
  - Toll processor for seal manufacturers
    - *Agreements in principle reached with partners*

Future Work Leading to Commercialization

- **FY05**
  - Understand effect of initial substrate roughness on UNCD friction
  - Commission and optimize 11-inch IPLAS system (up from 6-inch system)
    - *Demonstrate UNCD coating of multiple 2" seals simultaneously*
  - Demonstrate benefits of UNCD coatings on gas seals
  - Verify tribological benefits of UNCD coated seals
    - *Perform long-term pump tests*
- **FY06-07**
  - Pilot tests (field tests): 2" seals, 5" seals
  - Automation of UNCD deposition process
- **FY07**
  - Commercialization (sign toll-processing agreements)
  - Final report